



PATENT  
TH1848 (US)  
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE **RECEIVED**

**JAN 16 2004**

**TC 1700**

In re application of )

DAVID S. BROWN and )  
RICHARD E. ROBERTSON )

Serial No. 09/832,070 )

Filed April 10, 2001 )

NICKEL-CONTAINING ETHYLENE )  
OLIGOMERIZATION CATALYST AND USE )  
THEREOF )

) Group Art Unit: 1755

) Examiner: James W. Pasterczyk

) December 18, 2003

COMMISSIONER FOR PATENTS  
P. O. Box 1450  
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER RULE 132

David S. Brown, the undersigned, hereby declares:

THAT he is one of the coinventors of the above-identified United States patent application; and

THAT he has reviewed the literature article by Xue and believes that that paper and the present application teach fundamentally different points; that Xue teaches that the addition of a second phosphine or phosphite to the oligomerization system leads to a decrease in K-factor (lighter product distribution, i.e., more light olefins such as 1-butene and 1-hexene are formed), while the present application teaches that the addition of a phosphite with a concomitant reduction in a concentration of the active ligand leads to a system that gives the same activity and K-factor (and a heavier product distribution which is desired); and

THAT in 1989 he received a Bachelor of Science degree in Chemistry, Mathematics and English from Wake Forest University, and in 1993 he received a Ph.D. degree in Chemistry from the University of California, Berkeley; and

THAT since 1997, he has been employed by Shell Chemical LP and has worked on the Shell Higher Olefins Process and has supervised laboratory work related to the oligomerization of ethylene using nickel catalysts; and

THAT he provided the data which appears in Table 1 of the Response to Rejection dated October 7, 2003; and that he understands that the Examiner has not accepted the arguments presented therein because the Examiner feels that N,N-dimethyl acetamide (DMA) is a water soluble Lewis base which reads on ingredient c) of the claims in this application; and

THAT referring to DMA as a base is accurate in the broad sense that all compounds can be classified as acids or bases; and that the water soluble bases specified for use in the present application are all good proton acceptors and that good proton acceptors are necessary in neutralizing the benzoic acid which is a decomposition product of the catalyst ligand; and that DMA is a relatively poor proton acceptor (weak base, pKa of 0.10 in water as described in Adelman's article in the Journal of Organic Chemistry, Volume 29, 1964, pages 1837-1844); and that the pKa of benzoic acid is about 4 and thus will not react with DMA; and that DMA can only be protonated by strong acids such as nitric acid; and that it is his expert opinion that DMA would not function as a base in the reaction system of the present invention; and

THAT in the reaction in the present application, the acid/base balance is very important in the commercial process; and that the process should run under slightly basic conditions because if the pH moves to the acidic side, the K-factor will decline and more light products will be formed; and that this situation is to be avoided because heavier products are the desired products in the present invention; and

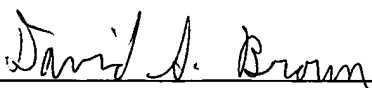
THAT the primary cause of acidity in the process of the present invention is the buildup of benzoic acid, which is a decomposition product of the catalyst ligand and thus, while the presence of benzoic acid is unavoidable, its disadvantages can be overcome by using a stronger base, such as potassium hydroxide, to help neutralize the benzoic acid; and

THAT DMA is a polar, aprotic solvent and that these properties make it a good solvent choice for boron hydrides (such as  $\text{NaBH}_4$ ) because the hydrides are strong bases themselves; and thus, hydrides can react with polar, protic solvents (such as water) to produce hydrogen gas, which is obviously an undesirable side reaction; and, for these reasons, in the lab DMA is a common solvent for boron hydride; and that in a commercial process, water is more advantageously used as a solvent because it is much less expensive than DMA; and that the poor stability of the  $\text{NaBH}_4$  in water can be overcome by using, a stronger base, such as potassium hydroxide, than DMA; and

THAT from a commercial process standpoint DMA is a poor solvent choice due to both its cost (relative to water) and its toxicity.

David S. Brown further declares that all statements herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date 12-19-03

  
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David S. Brown